DK 00/348   #2

REC'D 19 JUL 2000
| WIPO | PCT |

4

Kongeriget Danmark

Patent application No.:   PA 1999 00918

Date of filing: 28 June 1999

Applicant: Forskningscenter Risø
Postboks 49
DK-4000 Roskilde

This is to certify the correctness of the following information:

The attached photocopy is a true copy of the following document:

-   The specification, abstract, claims and drawings as filed with the application on the filing date indicated above.

**PRIORITY
DOCUMENT**
SUBMITTED OR TRANSMITTED IN
COMPLIANCE WITH RULE 17.1(a) OR (b)

**Patent- og
Varemærkestyrelsen**
Erhvervsministeriet

TAASTRUP 12 July 2000

Lizzi Vester
Head of Section

NANOMETER-SCALE MODULATION

FIELD OF THE INVENTION

5   The present invention relates to techniques for fabricating artificial periodic structures in crystals and at crystal interfaces and surfaces. In particular the present invention relates to techniques for fabricating nanometer-scale periodic strain patterns near the interface between two crystals, where the strain extends some distance away from the interface.

10

The present invention further relates to the use of such strain patterns to generate a regular lattice of quantum dots or quantum wires for applications in electronic, optoelectronic and magnetic device fabrication. The present invention even further relates to the use of such strain patterns to generate a regular nanoscale lattice of controlled

15   period at or near the free surface of a crystal for applications in diffractive optical systems, metrology and crystal growth.

BACKGROUND OF THE INVENTION

20   The most common way of making artificial periodic structures with nanometer-scale periods on crystals is by lithographic techniques such as photolithography and electron beam lithography. Such periodic structures are of technological interest for a variety of applications in electronics and optoelectronics, especially on semiconductor crystals, in storage systems, especially magnetic storage, using magnetic materials, and in the

25   production of diffraction grating, typically made of metallic materials.

However, lithographic techniques have serious limitations. Optical lithography has limitations mainly in terms of the smallest feature sizes that can be generated, which due to diffraction effects are not smaller than half the wavelength of the light used, and in

30   practice are considerably larger than this diffraction limit. For example, grating like modulation of GaAs/AlGaAs quantum wells has been achieved by use of an interference pattern from a laser, but the period of the fringes was limited to 2 μm using 532 nm laser light.

Electron beam lithography can be used to make features with smaller periods. However, the technology is slow if large areas on a surface must be patterned. And the regularity of the periodic pattern can be limited by aberrations of the electron optics. For example, Messica et al. describe an array of 25 metallic wires with a period of

5   100 nm formed by e-beam lithography.

In US patent US 5,532,510 a method is proposed for making periodic strain patterns by generating 10 nanometer-wide apertures by electron or ion beam lithography, but the issues of pattern regularity and processing speed are not addressed.

10

An alternative method for producing large scale, highly regular periodic patterns near crystal surfaces has been presented by Rocke et al., and involves the use of surface acoustic waves to modulate the strain of the crystal.

15 It is a disadvantage that this method is limited by the wavelength of the surface acoustic waves that can be generated (typically several microns), and is in any case cumbersome, as it requires external power to maintain the modulation.

Low-dimensional systems in optoelectronics, and in particular towards the fabrication

20 of so-called quantum wires and quantum dots have recently gained a lot of interest. Quantum wires and dots have dimensions in the range of 1-100 nm to provide sufficient confinement of electrons so that the desired quantisation effects are detectable at room temperature. Quantum wires induced by periodic strain have been made by the technique of cleaved-edge overgrowth.

25

It is a disadvantage that the above-mentioned technique is based on growing alternating layers of different materials by molecular beam epitaxy, which is rather cumbersome for the production of structures with more than a few periods.

30 It is a further disadvantage that there is no obvious way to extend the above-mentioned technique to fabricate regular arrays of quantum dot structures.

There is an extensive literature on the formation of arrays of quantum dot structures by so-called Stranski-Krastanov growth, whereby an overgrown material spontane-

ously forms nanometer scale islands on a surface, rather than growing in a layer by layer fashion. For example, Sopanen et al. have used the elastic strain induced by such islands to modulate the electronic structure of a semiconductor layer buried beneath the surface. The limitations of this technique for forming regular periodic struc-

5 tures are the lack of control over the regularity and periodicity of the array of islands. Multilayer techniques may improve the uniformity of the size and spacing of the islands, as described by Tersoff et al. and in US Patent US 5,614,435. However, these techniques result in something that is still very far from the perfection of a single crystal lattice. Another approach to making regimented arrays using electrochemical

10 techniques to form arrays of etch pits on which the islands nucleate is described in US 5,747,180. But again, regularity is only achieved in a statistical sense.

Bonding of crystal wafers has been used to form substrates that are suitable for epitaxial growth of other materials, as described in US Patent US 5,294,808. However,

15 US Patent 5,294,808 pays no attention to the use of any periodic structure generated at the bonded interface, and indeed depends on a lack of strain at the interface.

The strain at a bonded interface of two gold crystals has been investigated by Sass et al. and Taylor et al. However, these authors do not consider the possibility that this

20 strain may effect the electronic properties of the crystals, or the possibility of using this strain at the surface of a crystal to modulate materials grown on top of the crystal.

From the above discussion, it appears that no technique is currently available for fa-

25 bricating highly regular periodic structures over large areas of a crystal with periodicities in the range 1-100 nm. Furthermore, no attention has been drawn towards the used of such highly periodic structures.

It is an object of the present invention to utilise the periodically induced strain ex-

30 tending from an interface to modify the electronic and/or mechanical structure of a material in a controlled way so as to achieve a desired and technologically useful result.

It is a further object of the present invention to provide a technique for fabrication of highly regular periodic strain modulation in a crystal with periods in the range 1-100 nm.

5 It is a still further object of the present invention to provide a substantially periodic structure formed over large areas of a crystal interface by bonding of two crystal wafers.

It is an advantage of the present invention that the periodic strain structure can be
10 fabricated to extend some significant distance away from the interface, of order 1-10 nm, and that this depth is controllable.

It is a further advantage of the present invention that the regularity of the fabricated periodic structure, which is given by the regularity of the crystal lattices on either side
15 of the interface, may be extremely high.

SUMMARY OF THE INVENTION

The above-mentioned objets are complied with by providing, in a first aspect, a
20 method for providing a substantially periodic pattern, said method comprising the steps of:

- providing a first group of crystalline elements being formed by the same material and having a predetermined first crystal axis,

25

- providing a second group of crystalline elements being formed by the same material and having a predetermined second crystal axis being different from the first crystal axis, and

30 - bringing the first and second group of crystalline elements into contact with each other.

In a second aspect, the present invention relates to an article comprising:

- a first group of crystalline elements being formed by the same material and having a predetermined first crystal axis,

5

- a second group of crystalline elements being formed by the same material and having a predetermined second crystal axis being different from the first crystal axis,

10 wherein the first and second group of crystalline elements are adjacently positioned so as to form an interface region between the first and second group of crystalline elements, at least part of said interface region defining a substantially periodic pattern extending in at least one direction.

15 The material forming the first group of crystalline elements may comprise a semiconductor material, such as silicon or gallium arsenide. Similarly, the material forming the second group of crystalline elements may comprise a semiconductor material, such as silicon or gallium arsenide. Alternatively, the materials forming the first and second group of crystalline elements may comprise an insulator material, such as diamond or 20 sapphire.

The orientation of the crystal axes of the two wafers may differ in two different ways. There may be a twist angle between the crystal axes such that the in-plane axes of first crystal axis are rotated an angle θ relative to second crystal axis at the time of 25 bonding.

There may also be a tilt angle φ the crystal axes. This can be achieved by cutting the surface of one of the crystals at a tilt angle φ to the main crystal axes in the direction normal to the surface.

30

The tilt and twist of interest are typically in the range 0 degrees to 20 degrees, and in particular small angles in the range 0 degrees to 5 degrees. At larger angles, tilt is usually described in terms of the crystal facet index. At larger twist angles, the periodic interface structure is usually described in terms of coincidence site lattices.

The first crystal axis may differ from the second crystal axis by a twist angle θ, where θ may be within the range 0,1-10°, such as in the range 0,2-9°, such as in the range 0,3-8°, such as in the range 0,5-7°, such as in the range 1-5°.

5 Alternatively or in addition, the first crystal axis may differ from the second crystal axis by a tilt angle φ, where φ may be within the range 0,1-10°, such as in the range 0,2-9°, such as in the range 0,3-8°, such as in the range 0,5-7°, such as in the range 1-5°.

10 In the third aspect, the present invention relates to a laser comprising:

    - an article according to the second aspect of the present invention, the article further comprising:

15   - a material or a material system so as to form an array of quantum dots and/or quantum wires at or near the interface region, wherein the further material or material system distributes according to the substantially periodic pattern, and

20   - means for providing a pump signal for pumping the material or material system so as to emit electromagnetic radiation.

  The material or material system forming the array of quantum dots and/or quantum wires may comprise a semiconductor material, such as gallium, arsenide or indium or

25 any combination thereof. Furthermore, the material or material system forming the array of quantum dots and/or quantum wires has a thickness smaller than 200 nm, such as smaller than 150 nm, such as smaller than 100 nm, such as smaller than 80 nm, such as smaller than 50 nm.

30 The material or material system forming the array of quantum dots and/or quantum wires may be overgrown with an additional material, such as silicon or gallium arsenide.

The pump signal may comprise electromagnetic radiation in the radio frequency, visible or near-infrared range. The pump signal may also comprise a direct or alternating electric current passed through the quantum dots to stimulate the emission of radiation. The emitted electromagnetic radiation may be in the visible or near-infrared range.

5

In a fourth aspect, the present invention relates to an object for calibrating an instrument, said object comprising:

- an article according to the second aspect of the present invention, wherein the
10   substantially periodic pattern is transferred to a surface of the article.

The transferring of the substantially periodic pattern to the surface may comprise removing at least part of the first or second group of crystalline elements by etching. Alternatively, the transferring of the substantially periodic pattern to the surface may
15 comprise removing at least part of the first or second group of crystalline elements by chemical-mechanical polishing.

The transferred substantially periodic pattern is adapted to hold an additional material, such as a metal.

20

In the fifth aspect, the present invention relates to an element for splitting an incoming beam into one or more outgoing beams, said element comprising:

- an article according to the second aspect of the present invention, wherein the
25   incoming beam is incident on at least part of the substantially periodic pattern, said incoming beam having a first propagating direction, and wherein the one or more outgoing beams are reflected or transmitted by at least part of the substantially periodic pattern in one or more propagation directions being different from the first propagation direction.

30

In a sixth aspect, the present invention relates to an object for magnetically storing information, the said object comprising

- an article according to the second aspect of the present invention, wherein the substantially periodic pattern is transferred to a surface of the article, said transferred substantially periodic pattern being adapted to hold a plurality of magnetic structures so as to form a plurality of magnetic domains.

5

The plurality of magnetic structures may comprise iron, cobalt, chromium or any combination thereof. The plurality of magnetic structures are arranged according to the substantially periodic structure. The plurality of magnetic structures may be overgrown with a non-magnetic material.

10

BRIEF DESCRIPTION OF THE DRAWINGS

Fig. 1 illustrates the displacement field near the interface of two crystals bonded at their (001) surfaces at a twist angle θ. (A) The bonding produces atomic displace-

15   ments within a layer of characteristic thickness t. (B) Grid illustrating the modulation of the atom positions in a layer perpendicular to the interface. (C) The two atomic layers on either side of the interface, illustrating the dislocation network of period d. The model described in the following section has been used to calculate the atom positions in (B) and (C). The line ss in (C) shows where the layer in (B) cuts through the

20   interface.

Fig. 2. (A) The reciprocal lattice in a plane parallel to the interface. P_1 and P_2 are bulk Bragg $(1\bar{1}1)$ reflections and the cross indicate a satellite reflection. (B) The satellite reflection S in (A) scanned along the in-plane direction [h,h,0] at I = 1, for a sample

25   with a twist angle of 7.45°. The full width half maximum of the reflection is $\Delta h = 1.5$ 10^{-4} in reciprocal lattice units (r.l.u.). (C) A series of scans through the satellite reflection S at I = 1, measured along the out-of-plane direction I, for twist angles between 0.41° and 7.45°. The abscissa ΔI measures the distance from the satellite point at I = 1. The solid curves are fitted Lorentzian-squared functions. A measured background

30   intensity has been subtracted from all data points and the individual curves have been shifted vertically for clarity.

Fig. 3. Open circles are the measured full width half maximum values w for scans along the out-of-plane direction I (a subset is shown in Fig. 2C) as a function of the twist angle θ. The full line is calculated using the screw dislocation model described in the text. The inset shows the same plot on a linear scale.

Fig. 4 shows the calculated displacements dy of atoms along the x direction in the interface plane, given relative to the unperturbed crystal far away from the interface at different layers z from the interface, for a twist angle of θ = 4.4°. The open circles are calculated with the screw dislocation model described in the text. The solid lines are fitted sine functions. The amplitude of the sine function decays exponentially with z. The curves are shifted vertically for clarity.

DETAILED DESCRIPTION OF THE INVENTION

Periodic elastic modulation of a semiconductor crystal on the nanometer scale gives rise to novel electronic and optical properties. For example, external elastic modulation of a buried quantum well can lead to the formation of quantum wires or quantum dots.

Several methods have been developed to introduce a lateral periodic elastic modulation in the subsurface region of a semiconductor: cleaved edge overgrowth on a multiple quantum well, subsurface strain due to self-organised nanoscale islands or surface acoustic waves. Each of these techniques has limitations in either the extent, regularity or temporal stability of the periodic modulation that can be produced. Alternative methods to achieve lateral periodic elastic modulation extending a significant depth into a semiconductor are therefore of considerable interest.

Fusion bonding of two semiconductor wafers, where the orientation of one wafer is twisted with respect to the other by an angle θ (see Fig. 1A) results in the formation of a regular network of screw dislocations at the interface (see Fig. 1B,C), as observed by transmission electron microscopy. The lattice spacing d of the dislocation network is given by:

$$d = \frac{(a/\sqrt{2})}{2\sin(\theta/2)} \qquad (1)$$

where a = 5.43 Å is the lattice constant of silicon, and a/√2 is the nearest-neighbor interatomic distance in the (001) interface plane. Such artificial twist grain boundaries
10 have been studied extensively for gold bicrystals. Bonded semiconductor wafers have been studied at large twist angles ($\theta > 10°$), where thin crystals become elastically compliant, indicating that the elastic perturbation of one crystal by the other is small.

The present invention focuses on small twist angles. It is demonstrated that the cha-
15 racteristic thickness t of the elastic modulation, defined as the sum of the exponential decay lengths of the modulation amplitude to either side of the interface (see Fig. 1), is inversely proportional to the twist angle θ.

The technique used to investigate the elastic modulation near the interface is synchro-
20 tron X-ray diffraction. The penetration of high intensity X-rays enables the non-de-structive structural investigation of buried interfaces. The samples were prepared by direct wafer bonding, without an intermediate adhesive or oxide layer, which is a well-established technique for a wide variety of applications in microelectronics and mi-cromechanics. The wafers used were 10 cm diameter commercial grade mirror-po-
25 lished Si(001), 350 μm thick, and were stripped of their native oxide in a 5% HF solu-tion prior to contacting in a class 100 clean-room. The contacted pairs were then an-nealed at 1000°C for 1 hr in a nitrogen atmosphere to achieve high-strength silicon covalent bonding at the interface. Samples (~1 cm²) diced from wafer pairs that were bonded with twist angles between 0.4° and 7.5°. The tilt angle (miscut) of the wa-
30 fers, defined as the misorientation of the physical surface relative to the crystallo-graphic [001] direction, was less than 0.1°. To reduce X-ray absorption, one of the two bonded wafers was thinned to about 30 μm by mechanical grinding followed by chemical etching. The X-ray measurements were performed at the undulator beamline ID32 at the European Synchrotron Radiation Facility (ESRF) and at the wiggler beam-
35 line BW2 at HASYLAB, using six circle diffractometers.

The wavelengths used were 0.5254 Å and 1.24 Å at ID32 and HASYLAB, respec-
tively. Must of the experiments were performed at ID32, only one sample, where one
of the wafers was thinned to 1.5 μm was measured at BW2. The typical beam size at
sample at ID32 was 0.5mm vertical, 0.2mm horizontal. The samples were mounted in

5 air with sample surface normal aligned with the ω - rotation axis. The angle α between
incoming x-ray beam and sample surface was kept constant during scans, typically α
= 3°, giving a typical exit angle β = 2.5°. For these angles and the quoted sample
thickness, the total beam attenuation is about 30%. The detector slits before the
sample was typically 0.5 x 0.5 mm². The intensities along satellite rods were meas-

10 ured by measuring the peak and subtracting the average background from either side
of the peak. Several rods were measured using the conventional ω-integration and all
gave the same results as obtained when both peak signal and background were mea-
sured more simply by scanning parallel to the rod.

15 A superstructure of period, d, confined to a region near the bonded interface results in
an array of Bragg reflections in the reciprocal space probed by X-ray diffraction. These
reflections are elongated in the direction normal to the interface and spaced by $2\pi/d$ in
the plane of the interface. For a nearly harmonic displacement of the atomic positions,
only the satellite reflections closest to the bulk Bragg reflections of the two crystals

20 (see Fig. 2A) have significant intensity. Conventional cubic reciprocal lattice notation
is used. The width Δh of these satellite reflections along the in-plane [h,h,0] direction
in reciprocal space is inversely proportional to the lateral coherence length ξ of the pe-
riodic superstructure, $\xi = (a/\sqrt{2})/\Delta h$.

25 Fig. 2B shows a scan through a satellite reflection S close to the $(\bar{1}\bar{1}1)$ Bragg reflec-
tion of a bonded wafer pair with $\theta = 7.45°$. The peak width is $\Delta h = 1.5 \times 10^{-4}$ in reci-
procal lattice units and is resolution limited. From this value a lower limit to the cohe-
rence length of $\xi > 2\mu m$ is deduced. This is much greater than the typical terrace
widths on a free surface, which are ~1000 Å for a miscut of 0.1°. This implies that

30 the long-range order of the dislocation network is determined by the perfection of the
bulk crystals rather than their surfaces.

The satellite reflections closest to the $(1\bar{1}1)$ reflections were scanned along the out-of-plane direction for a set of seven bonded wafer pairs with different twist angles. Four examples are shown in Fig. 2C. The line shape of the measured reflections is fitted with a Lorentzian-squared function:

$$I(\Delta l) = I_0 \left(\frac{b^2}{b^2 + \Delta l^2} \right)^2 \qquad (2)$$

10 where the full width at half maximum is $w = 2b\sqrt{\sqrt{2}-1}$ and Δl is the distance to the satellite point at $l = 1$. For example, for $\theta = 0.41°$, a fit to the data in Fig. 2C of a Lorentzian line shape with p, the power of the Lorentzian, as a fit parameter, gives p $= 2.07 \pm 0.10$. A Lorentzian-squared function is the Fourier transform of a double-sided exponential decay function, in other words the elastic modulation decays exponentially to both sides of the interface. The characteristic thickness t is related to w

15 by $t = \dfrac{a}{\pi b}$. Thus, as the characteristic thickness t of the periodically modulated region increases the satellite Bragg peaks sharpen along the l-direction, as shown in Fig. 2C.

In Fig. 3, the measured width of these peaks is plotted against the twist angle θ,

20 which can be accurately measured from the angular separation of the bulk Bragg peaks of the two crystals. A linear relationship between w and θ is observed, which implies that t diverges as θ tends to zero.

The results are summarized in table 1 where the double exponential decay length t of

25 the displacement field, as derived from the measured diffraction profiles perpendicular to the interface, is listed. The full width half maximum w is measured in reciprocal lattice units. The twist angle θ and period d are shown in Fig. 1.

θ	d (Å)	w (r.l.u.)	t (Å)
0.41° ± 0.05°	230	0.014 ± 0.001	159
1.34° ± 0.07°	164	0.039 ± 0.003	58
1.40° ± 0.12°	157	0.050 ± 0.005	45
3.76° ± 0.10°	58	0.147 ± 0.010	14
4.41° ± 0.10°	50	0.20 ± 0.02	11
5.74° ± 0.10°	38	0.19 ± 0.02	12
7.45° ± 0.15°	29	0.34 ± 0.02	6.5

Table 1

In order to interpret these results quantitatively, the displacement field has been
5 simulated numerically for two simple cubic lattices, forming a square network of dislo-
cations at their common interface. Using an analytical expression for the displacement
field of a screw dislocation in a cubic lattice, the atomic displacement pattern due to a
square array of screw dislocations spaced by $N = d/a$ lattice parameters have been
calculated. Assuming the crystal is elastically isotropic, the result is independent of
10 the specific elastic constants of the crystal.

Fig. 4 shows the lateral modulation, Δy, of the atomic positions along the in-plane x-
direction of one of the cubic lattices for $N = 13$ corresponding to $\theta = 4.4°$, for diffe-
rent atomic layers at distance z from the interface.
15

In the simulations, the amplitude of the modulation of atomic positions decays expo-
nentially with depth from the interface over several decades, in agreement with ex-
periment. Further, the characteristic thickness t deduced from these simulations, when
plotted as a corresponding peak width, agrees quantitatively with measurements (full
20 line in Fig. 3), confirming the validity of this simple model. For Eqn. 1 in the small an-
gle limit, d is inversely proportional to θ, and so proportional to t. Although one might
expect a higher density of screw dislocations to lead to larger displacements of atoms
from their equilibrium positions and hence a larger depth of the modulation, in fact the
opposite is observed. This can be understood qualitatively by noting that, at points
25 between two parallel screw dislocations, the displacement field due to each screw
dislocation has opposite sign and tends therefore to cancel out. Thus at large twist

angles, the effect of the dislocation network is strongly localized, and long-range elastic interaction between the crystal lattices is suppressed, consistent with the behavior of compliant substrates bonded at large twist angles.

5 In contrast, at small twist angles, the elastic modulation can be made to extend many nanometers into the silicon wafers. Indeed, the modulation penetrates much further into the crystals than the characteristic thickness t, due to the long exponential tails.

This effect could be used to modulate a buried quantum well in a semiconductor, re-
10 sulting in a highly ordered quantum dot structure. The dominant component of the strain at the interface is shear strain. For a twist angle of $\theta = 5°$ and at layer $z = 5$, a maximum shear strain of 1.5% is obtained from the above model. This is comparable to the strain amplitudes estimated for other modulation mechanisms. The elastically modulated region provides a regular lattice with periodicity in the range 1-10 nm, a
15 range for metrology applications that is difficult to access with lithographic techniques. In this case, the modulated region could be exposed by etching to within a few nanometers of the interface. This modulated substrate could also be used as a template for overgrowth of periodically strained layers.

20 It has been demonstrated that for a series of bonded Si(001) wafers with twist angles ranging from 0.4° to 7.5° the atomic displacement field decays exponentially away from the interface with a characteristic length inversely proportional to the twist angle. The strong diffraction signal observed at small twist angles from the superstructure of the crystals allows a detailed line shape analysis of the measured rod profiles.
25 The data strongly support a simple model for the interface structure, where the atomic displacements are determined by a square net of screw dislocations.
The results described in the present text are results for silicon (001) with a twist boundary. However, as the simulations show, the elastic modulation due to a dislocation array is not sensitive to the detailed elastic properties of the material involved, but
30 depends primarily on the spacing of the dislocations. A wide variety of materials can be successfully fusion bonded, including semiconductors such as GaAs, and insulators such as diamond and sapphire. Also, tilt boundaries will result in parallel rows of edge dislocations, with analogous consequences.

Thus, the results presented here can be generalized to a wide range of materials and crystallographic interfaces. Determination of atomic positions in the core of the dislocations require accurate form factor measurements including many higher order reflections which go beyond the present analysis, but is quite feasible for future studies.

5

In a first embodiment the periodic strain field may be used as a template for a meteorological calibration standard for Scanning Tunnelling Microscopy (STM), Atomic Force Microscopy (AFM) and Scanning Electron Microscopy (SEM) in the 3-100 nm regime. Calibration standards in this regime are difficult to obtain; the lattice constant

10 of standard crystalline materials are too small and most artificial structures have too large a lattice constant. A calibration standard can be accomplished by wafer bonding two Si(100) wafers with zero tilt at twist angles of θ, where θ is in the range 0.2°-7°. This results in a periodic dislocation network with lattice spacing given by equation (1).

15

In order to be used as a calibration standard this periodic lattice must be transferred to the surface of one of the two crystals. After bonding, one of the two crystals must be thinned down to 100 nm or less, in order to allow the periodic strain field in the direction perpendicular to the bonded interface to extend to the surface of the thinned

20 crystal. The thinning can be achieved by preparing one of the wafers with an H- implanted etch stop prior to bonding, and etching with standard etchants such as KOH to remove the silicon on one side of the interface down to the etch stop. As shown in table 1 the strain field has an exponential decay away from the interfaces with 1/e lengths of 1-30 nm, so the etch stop must have a comparable depth. Alternatively,

25 chemical-mechanical polishing techniques could be applied to thin one of the wafers. Alternatively, thin membranes could be produced in one of the wafers prior to bonding, by chemical or physical etching techniques such as reactive ion etching. Roughness of the surface defined by the etch stop due to etching can be reduced, for example, by repeated oxidation and etching in HF.

30

When the periodic strain field penetrates to the surface, it can be made more easily detectable, for example by overgrowth of a suitable material, such as a metal. A material is chosen which shows preferential growth at sites with a specific strain, leading to a replication of the periodic pattern of the strain field in the overgrown material. A

metallic overlayer would also have advantages for STM measurements, where the substrate must be conducting. The pattern will have the same periodicity as the underlying grain boundary, and can be imaged with the above mentioned scanning techniques. The period of the pattern is given by the twist angle between the two crystals

5 as described in equation (1), and can be measured independently and extremely accurately by, for example, X-ray techniques, in order to provide a standard reference for the scanning probe techniques. The twist angle can be determined with accuracy better than 0.01°. A single bonded wafer could produce hundreds of identical calibration standards for distribution by manufacturers of scanning probe systems to their cus-

10 tomers. Owing to the two dimensional nature of the periodic strain field, the calibration standards could also be used for calibrating distortions that affect the relative scales of perpendicular axes in the scanned images.

In a second embodiment the periodic strain field may be used as a template for

15 growth of quantum dot lasers. The efficiency of such lasers depend on being able to produce a very high density of clusters of semiconductor material on a surface, the cluster size being in the nanometer range, and the clusters being nearly monodisperse. The optical properties of such clusters can be compared to artificial atoms with sharp absorption lines, and this has a number of advantages for laser fabrication in, for ex-

20 ample, telecommunications applications. In particular, owing to the line spectra of such quantum dots, the wavelength of spontaneous emission of the dots is much more stable to temperature changes than so-called quantum wire or quantum well lasers. However, a key challenge in the fabrication of such dots by self organisation techniques has been the compromise between achieving a dense packing, and pre-

25 venting clustering.

Producing a periodic strain modulation at a free surface provides a way of controlling the cluster formation process by providing a periodic array of preferred nucleation sites as a template for overgrowth of quantum dots. The periodic lattice must be

30 transferred to the surface of one of the two crystals by the method described in the previous embodiment.

After bonding, one of the two crystals must be thinned down to about 100 nm, in order to allow the strain field in the direction perpendicular to the bonded interface to

extend to the surface of the thinned crystal. The thinning can be achieved by preparing one of the wafers with an H- implanted etch stop prior to bonding, and etching with standard etchants such as KOH to remove the silicon on one side of the interface down to the etch stop. As shown in table 1 the strain field has an exponential decay

5   away from the interfaces with 1/e lengths of 1-30nm, so the etch stop must have a comparable depth. Roughness of the surface defined by the etch stop due to etching can be reduced, for example, by repeated oxidation and etching in HF.

When the periodic strain field penetrates to the surface, any oxide or contamination
10 layer at this surface can be removed in vacuum by standard techniques such as high-temperature annealing and sputtering. Subsequently, deposition of semiconductor materials suitable for quantum dots, such as Indium Arsenide can be carried out, in a molecular beam epitaxy system or chemical vapour deposition system. Under suitable conditions of substrate temperature and deposition rate, the clusters will grow prefe-
15 rentially at specific sites on the strained surface, which minimise the strain energy between the overgrown quantum dots and the substrate. In this way, a periodic pattern of quantum dots can be made. By varying the twist angle of the semiconductor wafers prior to bonding, the spacing of the quantum dots can be controlled, to achieve optimum density for a given mean quantum dot size. By varying the material the wa-
20 fers are made of (e.g. silicon, gallium arsenide) the electronic state of the quantum dots, and hence their optical properties, can be controlled. After preparation of the quantum dots, a protective cap layer of semiconductor material can be deposited on the quantum dots to protect them from oxidation. Further electrical connections to provide injection of carriers can be fabricated by prior art methods. Injection of charge
25 carriers is necessary to stimulate lasing.

In a third embodiment the periodic strain field may be used as a modulator of a buried quantum well layer, in order to form a quantum dot laser (described above). In this embodiment, it is not necessary to thin one of the wafers very accurately. Instead,
30 prior to bonding, a quantum well structure is fabricated in one of the wafers, with a suitable thin protective layer. Such a quantum well structure consists of a nanometer-thickness layer of one type of semiconductor grown epitaxially on another, for example gallium indium arsenide on gallium arsenide, using standard techniques such as molecular beam epitaxy. The thickness of the thin protective layer is 100nm or less,

such that the periodic strain field generated at the interface between the two wafers can penetrate through the protective layer and modulate the quantum well. This strain modulation will have the effect of modulating the electronic band-structure of the charge carriers in the, creating periodic electronic structure analogous to an array of

5   quantum dots.

In prior art, such sub-surface modulation has been produced by growth of clusters at a free surface. This embodiment provides a more controlled technique, where by varying the twist angle of the semiconductor wafers prior to bonding, the spacing of the quan-

10 tum dots can be controlled, to achieve optimum density for a given mean quantum dot size. After preparation, further electrical connections to the modulated quantum well can be fabricated by prior art. These provide a means to inject charge carriers in order to stimulate lasing.

15 In a fourth embodiment the periodic strain field may be used as a template for growth of arrays of periodic magnetic nanostructures, for applications such as data storage. An important challenge in data storage materials is the fabrication of regular magnetic nanodomains that can be addressed individually. Self-organisation techniques for achieving this depend on a compromise between achieving a dense packing, and pre-

20 venting clustering.

Producing a periodic strain modulation at a free surface provides a way of controlling the cluster formation process by providing a periodic array of preferred nucleation sites as a template for overgrowth of magnetic nanostructures. The periodic lattice

25 must be transferred to the surface of one of the two crystals by the method described in the first embodiment.

Again, after bonding, one of the two crystals must be thinned down to about 100 nm, in order to allow the strain field in the direction perpendicular to the bonded interface

30 to extend to the surface of the thinned crystal. The thinning can be achieved by pre-paring one of the wafers with an H- implanted etch stop prior to bonding, and etching with standard etchants such as KOH to remove the silicon on one side of the interface down to the etch stop. As shown in table the strain field has an exponential decay away from the interfaces with 1/e lengths of 1-30nm, so the etch stop must have a

comparable depth. Roughness of the surface defined by the etch stop due to etching can be reduced, for example, by repeated oxidation and etching in HF.

When the periodic strain field penetrates to the surface, any oxide or contamination
5 layer at this surface can be removed in vacuum by standard techniques such as high-temperature annealing and sputtering. Subsequently, magnetic materials are deposited suitable for data storage, such as iron, cobalt, chromium, and alloys of such materials, using techniques such as molecular beam epitaxy system or chemical vapour deposition system. Under suitable conditions of substrate temperature and deposition rate,
10 the clusters will grow preferentially at specific sites on the strained surface, which minimise the strain energy between the overgrown magnetic nanostructures and the substrate. Optionally, a buffer layer of one metallic material may be deposited prior to the active magnetic material, in order to optimise diffusion of the magnetic material. In this way, a periodic magnetic pattern can be made. By varying the twist angle of the
15 semiconductor wafers prior to bonding, the spacing of the quantum dots can be controlled, to achieve optimum density for a given mean quantum dot size.

ABSTRACT

A new method to artificially modulate a crystal lattice is disclosed, where the modulation has a controlled periodicity and thickness in the range of one to several hundred

5 nanometers. The present invention relates to the fabrication of periodically strained crystal lattices where the period and thickness of the modulated region is controlled by bonding two crystal wafers at a specified twist angle. Two polished and clean crystal wafers are placed in intimate contact at a specified twist angle and, if necessary, heated to obtain bonding between the two wafers. The two crystal lattices

10 modulate each other, resulting in a modulation near the interface between the crystals with a periodicity that is different from that of the crystal lattices. This periodic modulation affects the electronic and structural properties of the two crystals in the vicinity of the interface. The modulation of the electronic properties leads to the formation of a highly regular quantum dot or quantum wire structures of controlled period

15 near the crystal interface, with applications in electronics, optoelectronics and magnetic devices. The modulation of the crystal structure can be exploited for the formation of metrological standards in the 1-100 nanometer range, or gratings for diffractive optic elements with grating periods in the same range. By transferring the periodic modulation to the free surface of a crystal, it can be used as a template for further

20 growth of periodically modulated crystalline layers by evaporation techniques.

22769DK1/STM/28-06-99

CLAIMS

1. A method for providing a substantially periodic pattern, said method comprising the steps of:

5

    - providing a first group of crystalline elements being formed by the same material and having a predetermined first crystal axis,

    - providing a second group of crystalline elements being formed by the same material and having a predetermined second crystal axis being different from the first crystal axis, and

10

    - bringing the first and second group of crystalline elements into contact with each other.

15

2. A method according to claim 1, wherein the material forming the first group of crystalline elements comprises a semiconductor material, such as silicon or gallium arsenide.

20 3. A method according to claim 1 or 2, wherein the material forming the second group of crystalline elements comprises a semiconductor material, such as silicon or gallium arsenide.

4. A method according to claim 1, wherein the materials forming the first and second

25 group of crystalline elements comprise an insulator material, such as diamond or sapphire.

5. A method according to any of claims 1-4, wherein the first crystal axis differs from the second crystal axis by a twist angle θ, wherein θ is within the range 0,1-10°, such

30 as in the range 0,2-9°, such as in the range 0,3-8°, such as in the range 0,5-7°, such as in the range 1-5°.

6. A method according to any of claims 1-4, wherein the first crystal axis differs from the second crystal axis by a tilt angle φ, wherein φ is within the range 0,1-10°, such as in the range 0,2-9°, such as in the range 0,3-8°, such as in the range 0,5-7°, such as in the range 1-5°.

5

7. An article comprising:

- a first group of crystalline elements being formed by the same material and having a predetermined first crystal axis,

10

- a second group of crystalline elements being formed by the same material and having a predetermined second crystal axis being different from the first crystal axis,

15 wherein the first and second group of crystalline elements are adjacently positioned so as to form an interface region between the first and second group of crystalline elements, at least part of said interface region defining a substantially periodic pattern extending in at least one direction.

20 8. An article according to claim 7, wherein the material forming the first group of crystalline elements comprises a semiconductor material, such as silicon or gallium arsenide.

9. An article according to claim 7 or 8, wherein the material forming the second group 25 of crystalline elements comprises a semiconductor material, such as silicon or gallium arsenide.

10. An article according to claim 7, wherein the materials forming the first and second group of crystalline elements comprise an insulator material, such as diamond or sap-
30 phire.

11. An article according to any of claims 7-10, wherein the first crystal axis differs from the second crystal axis by a twist angle θ, wherein θ is within the range 0,1-10°,

such as in the range 0,2-9°, such as in the range 0,3-8°, such as in the range 0,5-7°, such as in the range 1-5°.

12. An article according to any of claims 7-10, wherein the first crystal axis differs
5 from the second crystal axis by a tilt angle φ, wherein φ is within the range 0,1-10°, such as in the range 0,2-9°, such as in the range 0,3-8°, such as in the range 0,5-7°, such as in the range 1-5°.

13. A laser comprising:
10

- an article according to any of claims 7-12, the article further comprising:

- a material or a material system so as to form an array of quantum dots and/or quantum wires at or near the interface region, wherein the further ma-
15   terial or material system distributes according to the substantially periodic pattern, and

- means for providing a pump signal for pumping the material or material system so as to emit electromagnetic radiation.
20

14. A laser according to claim 13, wherein the material or material system forming the array of quantum dots and/or quantum wires comprises a semiconductor material, such as gallium, arsenide or indium or any combination thereof.

25 15. A laser according to claim 13 or 14, wherein the material or material system forming the array of quantum dots and/or quantum wires has a thickness smaller than 200 nm, such as smaller than 150 nm, such as smaller than 100 nm, such as smaller than 80 nm, such as smaller than 50 nm.

30 16. A laser according to any of claims 13-15, wherein the material or material system forming the array of quantum dots and/or quantum wires is overgrown with an additional material, such as silicon or gallium arsenide.

17. A laser according to any of claims 13-16, wherein the pump signal comprises electromagnetic radiation.

18. A laser according to claim 17, wherein the electromagnetic radiation is in the radio
5 frequency, visible or near-infrared range.

19. A laser according to any of claims 13-16, wherein the pump signal comprises a direct and/or alternating electric current.

10 20. A laser according to any of claims 13-19, wherein the emitted electromagnetic radiation is in the visible or near-infrared range.

21. An object for calibrating an instrument, said object comprising:

15   - an article according to any of the claims 7-12, wherein the substantially periodic pattern is transferred to a surface of the article.

22. An object according to claim 21, wherein the transferring of the substantially periodic pattern to the surface comprises removing at least part of the first or second
20 group of crystalline elements by etching.

23. An object according to claim 21, wherein the transferring of the substantially periodic pattern to the surface comprises removing at least part of the first or second group of crystalline elements by chemical-mechanical polishing.
25

24. An object according to claim 21, wherein the transferred substantially periodic pattern is adapted to hold an additional material, such as a metal.

25. An element for splitting an incoming beam into one or more outgoing beams, said
30 element comprising:

      - an article according to any of the claims 7-12, wherein the incoming beam is incident on at least part of the substantially periodic pattern, said incoming beam having a first propagating direction, and wherein the one or more outgoing

beams are reflected or transmitted by at least part of the substantially periodic pattern in one or more propagation directions being different from the first propagation direction.

5 26. An object for magnetically storing information, the said object comprising

- an article according to any of the claims 7-12, wherein the substantially periodic pattern is transferred to a surface of the article, said transferred substantially periodic pattern being adapted to hold a plurality of magnetic structures so

10 as to form a plurality of magnetic domains.

27. An object according to claim 26, wherein the plurality of magnetic structures comprise iron, cobalt, chromium or any combination thereof.

15 28. An object according to claim 26 or 27, wherein the plurality of magnetic structures are arranged according to the substantially periodic structure.

29. An object according to any of claims 26-28, wherein the plurality of magnetic structures are overgrown with a non-magnetic material.
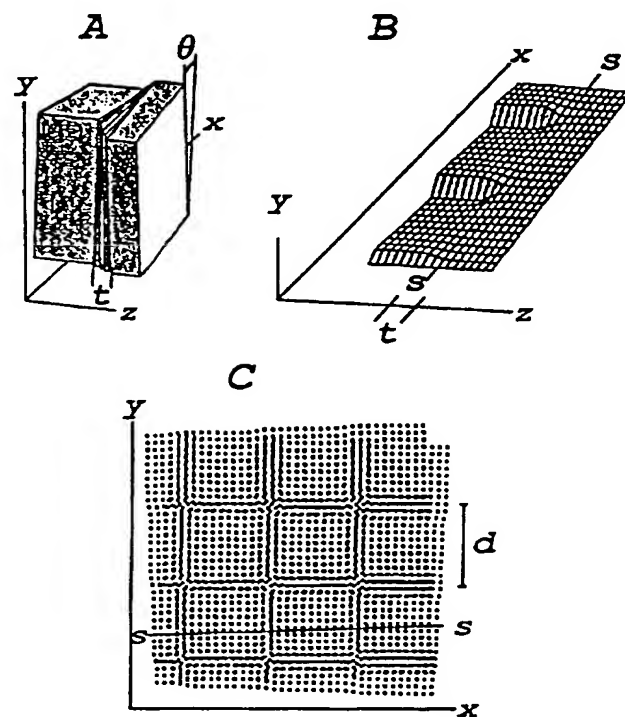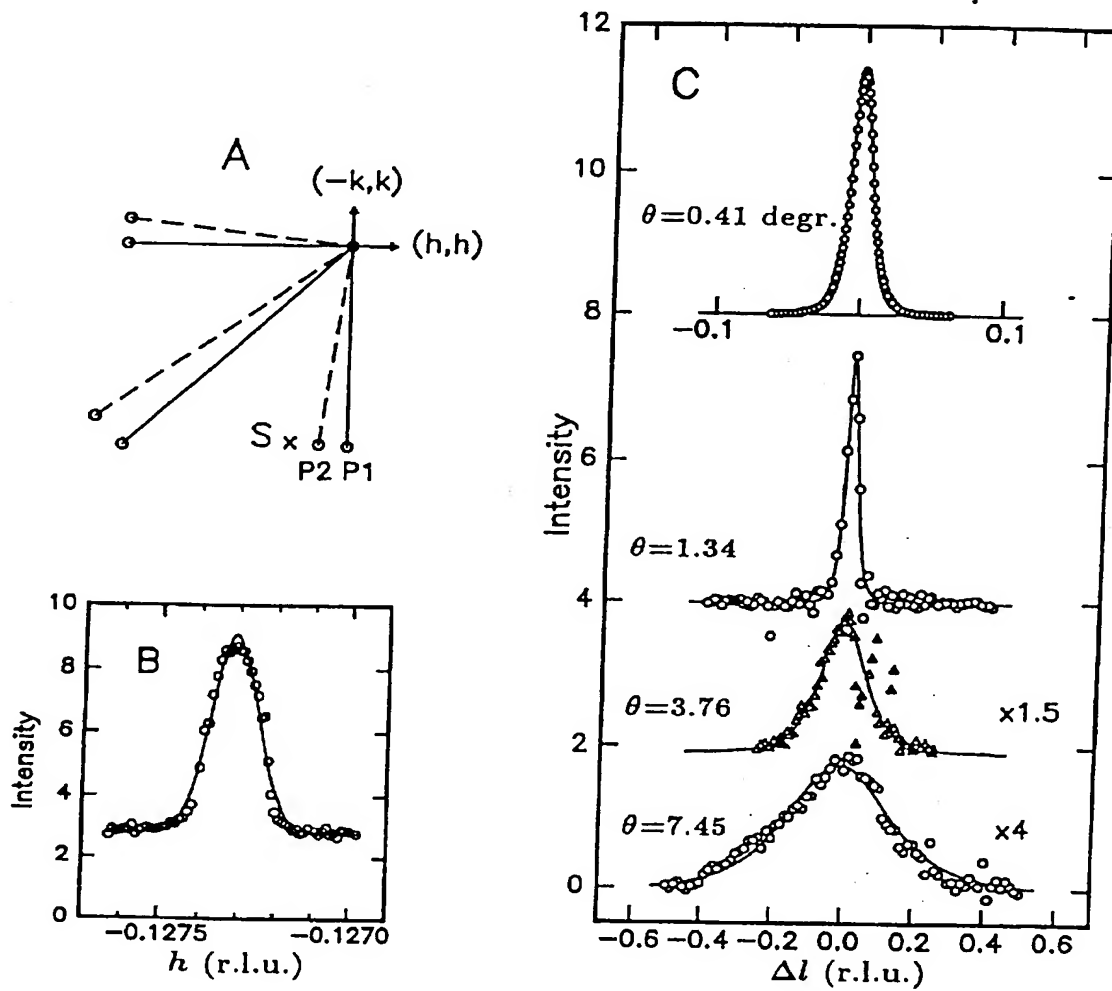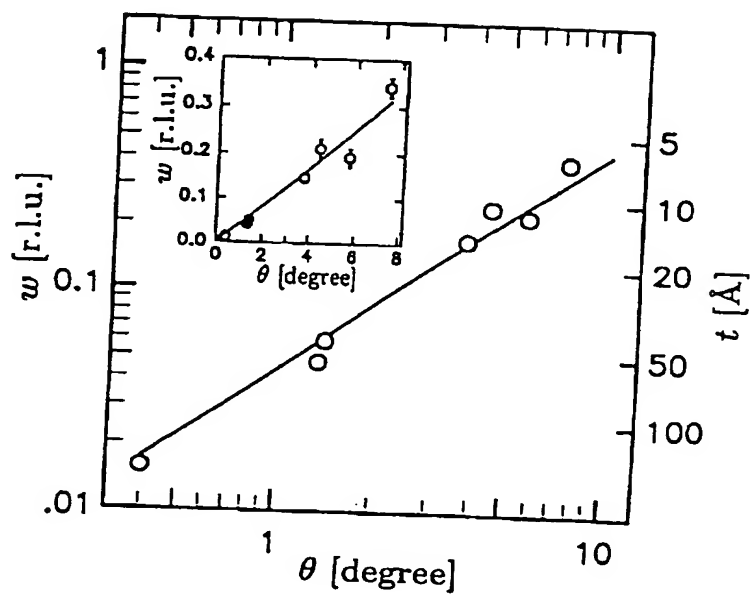
20

Fig. 1

Fig. 2

Fig. 3

Fig. 4